



*Mediterranean Archaeology and Archaeometry*, Vol. 10, No. 4, pp. 93-98

Copyright © 2010 MAA

Printed in Greece. All rights reserved.

## INVESTIGATION OF THE OSL SIGNAL FROM VERY DEEP TRAPS IN NATURAL QUARTZ

G. Kitis<sup>1</sup>, N.G. Kiyak<sup>2</sup>, G.S. Polymeris<sup>2,3</sup> and V. Pagonis<sup>4</sup>

<sup>1</sup>*Aristotle University of Thessaloniki, Nuclear Physics Laboratory, Thessaloniki, Greece*

<sup>2</sup>*ISIK University, Physics Department, Faculty of Science and Arts, 34980-Sile, Istanbul, Turkey*

<sup>3</sup>*Cultural and Educational Technology Institute-R.C. Athena/Archaeometry Laboratory, Xanthi, Greece*

<sup>4</sup>*McDaniel College, Physics Dept, Westminster, MD 21157, USA*

Received: 02/03/2010

Accepted: 15/06/2010

Corresponding author: [gkitis@auth.gr](mailto:gkitis@auth.gr)

---

### ABSTRACT

It has been recently reported by several studies that a thermally transferred optically stimulated luminescence (TT-OSL) signal from quartz grains can be used to extend the dating range for quartz samples. The TT-OSL signals are believed to consist of a recuperated OSL (ReOSL) component and a basic-transferred OSL (BT-OSL) component. In the present work the TT-OSL signals from several types of unfired quartz samples were studied. A special protocol was used, which allowed the measure the OSL from very deep traps (VDT) as a function of the OSL stimulation temperature. It was found that all quartz samples exhibit TT-OSL signals, which are depended on sample and on the OSL stimulation temperature. The activation energy of the process was evaluated and the influences of the TT-OSL on the ReOSL dating protocol are discussed.

---

**KEYWORDS:** Quartz, Deep traps, TT-OSL

---

## INTRODUCTION

Several recent studies have shown that a thermally transferred OSL (TT-OSL) signal from quartz grains can extend the dating range for quartz samples of various origins by almost an order of magnitude (Wang et al., 2006a; Wang et al., 2006b; Tsukamoto et al., 2008). It has been suggested that the TT-OSL signal comprises a recuperated OSL (ReOSL) component and a basic-transferred OSL (BT-OSL) component, Aitken (1998). Dating protocols based on the TT-OSL signal attempt to separate the BT-OSL signal and the ReOSL signal, by thermal treatments. It is suggested that the ReOSL signal is the major contributor to the TT-OSL signal. BT-OSL signal is a smaller but significant component. Aitken (1998) suggested that the ReOSL results from a "double transfer" process while the BT-OSL was due to electrons

trapped originally in light-insensitive traps. An alternative "single transfer" process for the production of ReOSL has been suggested by Adamiec et al. (2008) and was modeled by Pagonis et al. (2008). In the single transfer process, a light-sensitive trap acts as the source of electrons for the ReOSL. It is probable that the BT-OSL signal, or at least some part of it, originates from Very Deep Traps (VDT). As VDT we consider those traps which are responsible for TL glow-peaks with a peak maximum temperature of 500 °C.

The aim of the present work is to investigate the contributions of the OSL signals from the VDT in unfired natural quartz of various origins, to set protocols involving OSL measurements at high temperatures like ReOSL dating protocol.

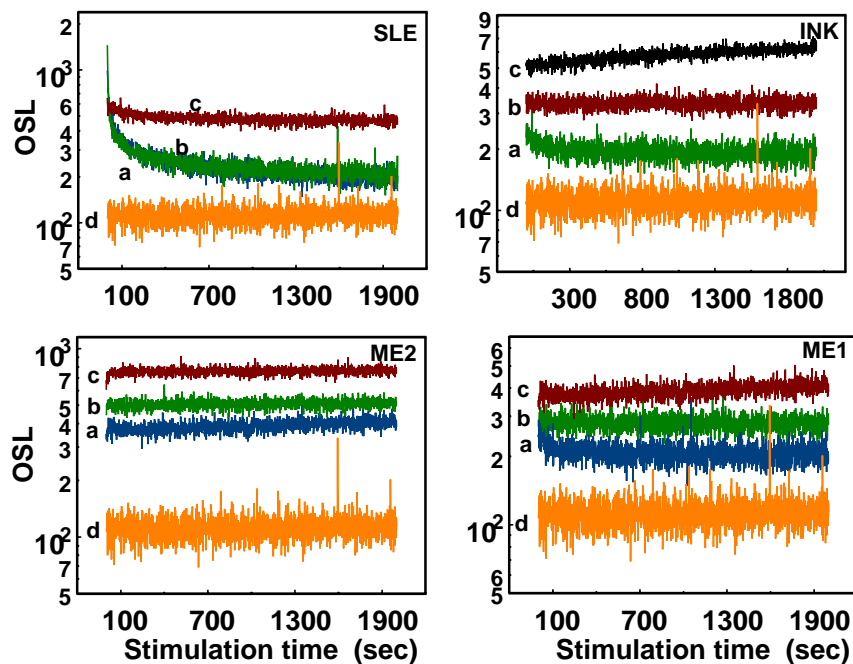


Figure 1: High efficiency TT-OSL curve shapes for quartz measured at various OSL stimulation temperatures. Curve (a) 190 °C, Curve (b) 250 °C and Curve (c) at 375 °C. Curve (d) corresponds to the background OSL signal measured at room temperature.

## MATERIALS AND APPARATUS

The samples were quartz of various origins and from diverse locations of Turkey. Samples with laboratory codes SLE and PDK were very young coastal-dunes near Istanbul, with 0.011 Gy and 0.008 Gy equivalent doses (Kiyak and Canel, 2006). Sample INK was also from young

coastal dunes from Inkumu- Bartın, in the middle part of the Black Sea Region with a natural dose 0.022 Gy. Sample PTR was a deposit from Patara in the Mediterranean coast and contained a natural dose about 2.2 Gy. Samples SRZ1 and SRZ2 were from different layers of a marine terrace in Saroz, on the Egean coastal area. These samples are old and the natural SAR pa-

leodose dose in quartz grains were estimated as 181 and 195 Gy respectively. ME1 and ME2 were from fluvial terrace sediments from an area close to the Marmara Ereglisi, in European part of the Marmara Region with 127 and 145 Gy paleodoses. The samples as received were wet sieved, and quartz grains with dimensions between 90 and 180  $\mu\text{m}$  were selected and deposited on stainless steel disks of 1  $\text{cm}^2$  area. Quartz purity of the samples was previously verified by IRSL measurements at ambient temperature.

Blue light emitting diodes (LEDs) (470 nm, 40  $\text{mW}/\text{cm}^2$ ) were used for stimulation intensities linearly increased between 0 and 36  $\text{mW}/\text{cm}^2$ . All measurements were performed using the RISØ TL/OSL reader (model TL/OSL-DA-15) equipped with about 0.1 Gy/s  $^{90}\text{Sr}/\text{Y}$   $\beta$ -ray source. The reader is fitted with an EMI 9635QA PM Tube. Luminescence signals were detected through U-340 filters. The TL readout

heating rate used was 1 K/s for all measurements.

## EXPERIMENTAL RESULTS AND DISCUSSION

The multi-aliquot protocol used to study the efficiency of TT-OSL from VDT was the following:

**Step 1:** Natural sample+300Gy beta dose.

**Step 2:** TL readout to 500  $^{\circ}\text{C}$ .

**Step 3:** Test dose (TD= 10 Gy) and TL to 500  $^{\circ}\text{C}$ .

**Step 4:** Blue CW-OSL at a variable stimulation temperature  $T_i$  (125C – 375C in steps of 25C) for 2000 s.

**Step 5:** Residual TL up to 500  $^{\circ}\text{C}$ .

**Step 6:** TD and TL to 500  $^{\circ}\text{C}$ .

**Step 7:** Repeat steps 1-6 for a new sample and new stimulation temperature  $T_i$ .

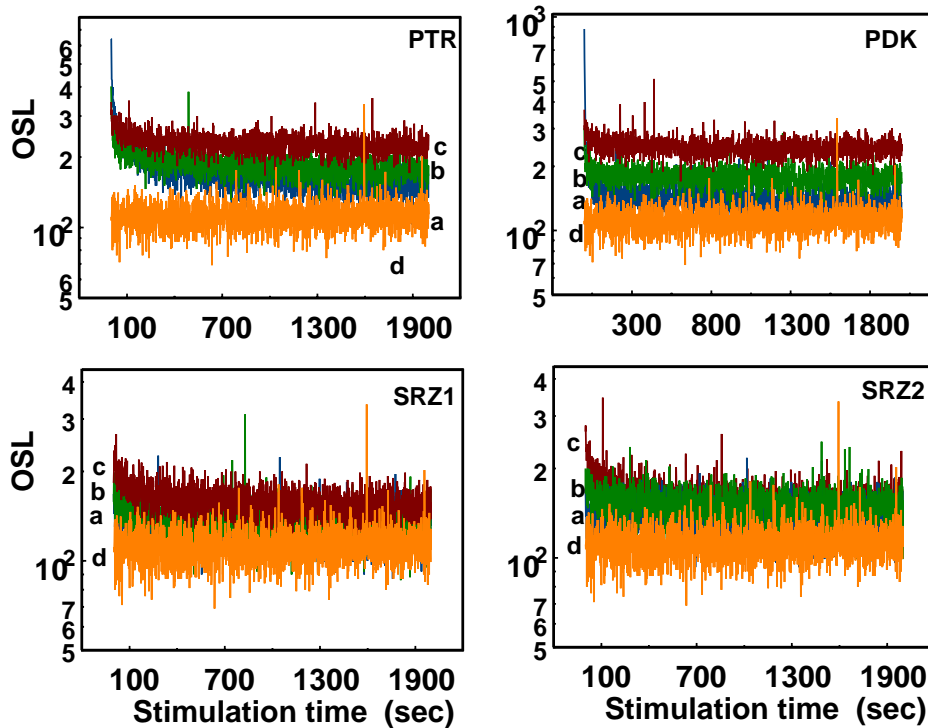


Figure 2: Low efficiency TT-blueOSL curve shapes for quartz measured at various OSL measuring temperatures. Curve (a) 190  $^{\circ}\text{C}$ , Curve (b) 250  $^{\circ}\text{C}$  and Curve (c) at 375  $^{\circ}\text{C}$ . Curve (d) corresponds to the background OSL signal measured at room temperature

Since the degree of filling of VDT by the geological dose is not known, an additional irradiation is given in step 1 to ensure the filling of the VDT. In step 2, any trapping level responsi-

ble for TL-glow-peaks up to 500  $^{\circ}\text{C}$  is erased. The measurement in step 3 is used to evaluate the sensitivity to a low test dose and verify that the heating up to 500  $^{\circ}\text{C}$  erases the TL signal

above 300 °C down to the background level. Step 4 aims to study the magnitude of the TT-OSL signal from VDT as a function of the OSL stimulation temperature. Step 5 is applied to find whether TL signal appears between 20–500°C after the stimulation at high temperatures, due, for example, to phototransfer process. Finally step 6 will evaluate the sensitivity to the low test dose as in step 3 and allows to evaluate how the applied protocol affects the sensitivity of the quartz sample.

The protocol was applied to the specific eight different quartz samples described above. In six of these samples only three OSL measuring temperatures were used (190, 250 and 375°C). On the other hand, since the OSL measurement at high temperatures is a thermally activated optical stimulation process, two samples were selected, one with high and another with low TT-OSL signals. In these two samples the optical stimulation was studied in the whole temperature region from 125 to 375 °C in steps of 25 °C. The purpose of this experiment is to evaluate the activation energy values of the thermally activated optically stimulated process.

The main result of this experiment responds directly to the first question set in the present work, namely to show that all quartz samples exhibit TT-OSL signals. The intensity of the OSL signals from VDT was found to depend on the type of quartz sample used. The TT-OSL curves from VDT are shown in Figs. 1 and 2. In Fig. 1 the samples having a very high efficiency for TT-OSL are presented whereas Fig. 2 shows the samples having low TT-OSL efficiency. In all cases the background level is included.

It is useful here to present in Fig. 3 the results of the steps 1, 2, 3, 5 and 6 of the protocol. The glow-curve (a) of Fig. 3 results after the irradiation with 300 Gy. The glow-curve (b) received in step 3 gives (i) the sensitivity to a low dose after the heavy irradiation in step 1 and assures (ii) the TL readout in step 2 erases the TL above 300°C to the usual background level and it does not leaves any intense residual TL signal at the very high temperatures. The absence of any signal in curve (c) of step 5, shows that the optical stimulation at the high temperatures of step 4 does not cause any re-trapping to the low temperature glow-peaks. Finally, the

glow-curve (d), which almost coincides with glow-curve (b) shows that the sample treatment in steps 2 to 5 does not influence the sensitivity of the samples.

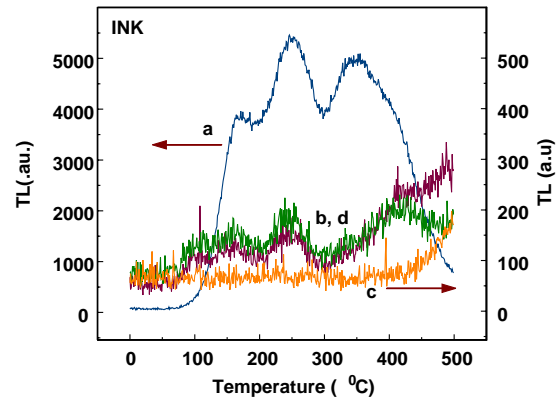


Figure 3: a) NTL plus 300 Gy beta dose. (b) TL glow-curve from step 3. (c) Residual TL after blue CW-OSL stimulation at the temperature of 375°C (d) TL from step 6.

The basic characteristic of TT-OSL curve is that the TT-OSL signal increases from zero to higher level and then takes the form of a more or less stable emission. However, in some samples for very short stimulation times a fast component appears. These cases are shown in Fig. 4, where only the first 300 s of the stimulation curves are included in the plot. Furthermore, it appears only for OSL stimulation temperatures lower than 175 °C, and not for higher stimulation temperatures. Obviously this component can not be related to the known fast component originating from the electron trap responsible for the 325 °C TL glow peak of quartz, since this electron trap was emptied by the TL readout up to 500 °C.

Fig. 5a shows the behavior of the total TT-OSL as a function of the OSL stimulation temperature. The exponential nature of the behavior allows the evaluation of the activation energy of the optical stimulation at high temperatures for two of the samples. The respective Arrhenius plots are shown in Fig. 5b. In the case of INK quartz sample which exhibits a strong TT-OSL signal, the activation energy was found to be  $E=0.44\pm0.03$  eV, whereas for the case of PDK quartz, which shows a weak TT-OSL signal, it was found to be  $E=0.42\pm0.04$  eV

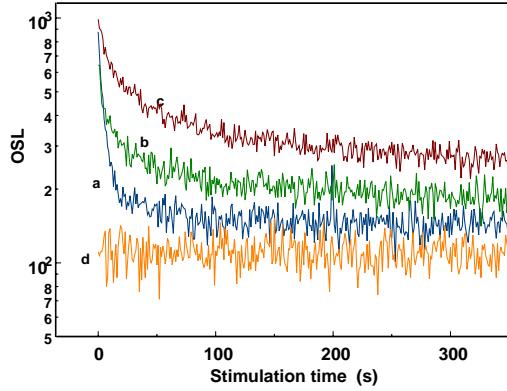


Figure 4: Examples of TT-OSL curves showing a fast component at very short stimulation times. Curve (a) PDK quartz, curve (b) PTR quartz, curve (c) SLE quartz and curve (d) the OSL background.

Since the thermal quenching effect is often reported in quartz, it is necessary to examine the results of Fig. 5 under the influence of thermal quenching. Assuming that the thermal quenching parameters  $C=2.8 \times 10^7$  and  $W=0.64$  eV, given by Wintle (1975) as representative for quartz, it is possible to correct the OSL intensities due to thermal quenching as follows. Initially, the values of the thermal quenching efficiency were evaluated for each one of the OSL stimulation temperatures.

Then the OSL intensity at each temperature is corrected due to thermal quenching by dividing it with the evaluated corresponding thermal quenching efficiency. Then the logarithm of the corrected OSL values is plotted against  $1/kT$

(Arrhenius plots). The results for both integrated regions are shown in Fig. 6. The resulting values of the activation energies were found to be  $E=1.02 \pm 0.03$  eV, and  $E=0.92 \pm 0.02$  eV for INK and PDK. Using only the present data, it is not possible to decide which values are the correct ones.

Therefore, separate experiments of TL as a function of heating rate are required in order to investigate if the thermal quenching effect is present or not in the specific quartz samples studied.

#### 4. CONCLUSIONS

Under the combined action of thermal activation and optical stimulation electrons trapped at VDT are liberated to the conduction band and recombine emitting photons. This is known as a TT-OSL signal. Eight unfired quartz samples of various origins that were studied, exhibited TT-OSL signals in a wide region of OSL stimulation temperatures. It was found that the TT-OSL signal depends strongly on the type of quartz sample studied.

This TT-OSL signal could be an appreciable component of the BT-OSL signal involved in the TT-OSL dating protocols. According to the results of the present work, the importance of the BT-OSL part of the TT-OSL dating protocol depends also on the sample studied, and we recommend that the BT-OSL signals are included in TT-OSL protocols, rather than being omitted.

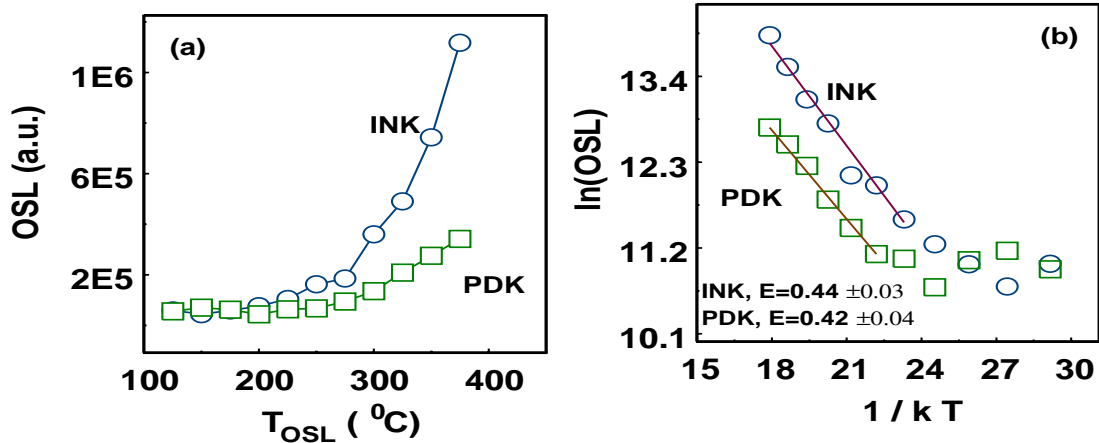


Figure 5: Arrhenius plot showing the activation energy evaluation for the TT-OSL signal without taking into account thermal quenching. INK quartz  $E=0.44 \pm 0.03$  eV and PDK quartz  $E=0.42 \pm 0.04$  eV

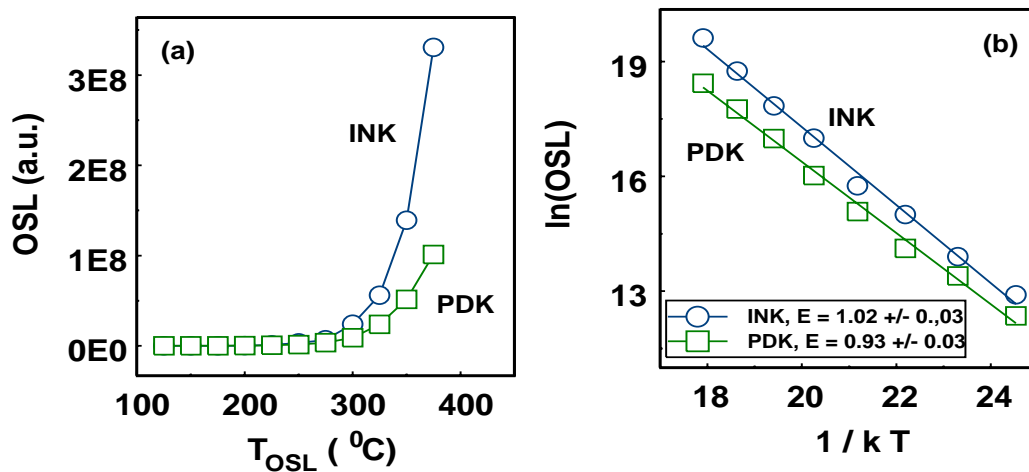


Figure 6: Arrhenius plot showing the activation energy evaluation for the TT-OSL signal taking into account thermal quenching. INK quartz  $E=1.02\pm0.03$  eV and PDK quartz  $E=0.92\pm0.02$  eV

## REFERENCES

- Adamiec, G.A., Bailey, R.M., Wang, X.L., Wintle, A.G., (2008). The mechanism of thermally-transferred optically stimulated luminescence. *J. Phys. D: Appl. Phys.* 41, 135503.
- Aitken, M.J., (1998). *An Introduction to Optical Dating*. Oxford University Press, Oxford.
- Bulur, E., (2000). A simple transformation for converting CW-OSL curves to LM-OSL curves. *Radiat. Meas.* 32, 141–145.
- Kitis, G. and Pagonis, V. (2008). Computerized curve deconvolution analysis for LM-OSL. *Radiat. Measur.* 43, 737–741.
- Kiyak, N.G., Canel, T., (2006). Equivalent dose in quartz from young samples using the SAR protocol and the effect of preheat temperature. *Radiat. Meas.* 41, 917–922.
- Pagonis, V., Wintle, A.G., Chen, R., Wang, X.L., (2008). A theoretical model for a new dating protocol for quartz based on thermally transferred OSL (TT-OSL). *Radiat. Meas.* 43, 704–708.
- Tsukamoto, S., Duller, G.A.T., Wintle, A.G., (2008). Characteristics of thermally transferred optically stimulated luminescence (TT-OSL) in quartz and its potential for dating sediments. *Radiat. Meas.* 43, 1204–1218.
- Wang, X.L., Lu, Y.C., Wintle, A.G., (2006a). Recuperated OSL dating of fine-grained quartz in Chinese loess. *Quat. Geochronol.* 1, 89–100.
- Wang, X.L., Wintle, A.G., Lu, Y.C., (2006b). Thermally-transferred luminescence in fine-grained quartz from Chinese loess: basic observations. *Radiat. Meas.* 41, 649–658.
- Wang, X.L., Wintle, A.G., Lu, Y.C., (2007). Testing a single-aliquot protocol for recuperated OSL dating. *Radiat. Meas.* 42, 380–391.